

1999DE119

A CLAIMS

Patent Claims

1. A process for the preparation of alkylphosphonous acid esters, which comprises
  - a) reacting elemental yellow phosphorus with alkylating agents in the presence of a base to give a mixture which comprises, as principal constituents, the (metal) salts of alkylphosphonous, phosphorous and hypophosphorous acids,
  - b) esterifying the principal constituents of the mixture from a) to give an ester mixture,
  - c) isolating the ester of the alkylphosphonous acid from the ester mixture.
- 15 2. A process as claimed in claim 1, wherein the alkylating agents are alkyl halides, dialkyl sulfates, trialkyl phosphates, dialkyl carbonates and/or formic acid ortho-esters.
3. A process as claimed in claim 1 ~~or 2~~, wherein the alkylating agent employed is methyl chloride, methyl bromide and/or dimethyl sulfate.
4. A process as claimed in ~~one or more of claims 1 to 3~~, wherein the bases are hydroxides, carbonates, bicarbonates, amides, alkoxides and/or amine bases.
- 25 5. A process as claimed in ~~one or more of claims 1 to 3~~, wherein the reaction in step a) is carried out in a two-phase system comprising aqueous alkali or

alkaline-earth metal hydroxide or mixtures thereof and an organic solvent.

A 6. A process as claimed in <sup>claim 1,</sup> ~~one or more of claims 1 to~~

A 5 ~~5,~~ wherein the organic solvents employed are straight-chain or branched alkanes, alkyl-substituted aromatic solvents, water-immiscible or only partially water-miscible alcohols or ethers, alone or in combination with one another.

A 7. A process as claimed in <sup>claim 1,</sup> ~~one or more of claims 1 to~~

A 10 ~~6,~~ wherein the organic solvent employed is toluene, alone or in combination with alcohols.

A 8. A process as claimed in <sup>claim 1,</sup> ~~one or more of claims 1 to~~

A ~~7,~~ wherein the reaction is carried out in the presence of a phase-transfer catalyst.

A 15 9. A process as claimed in claim 8, wherein the phase-transfer catalyst is a tetraalkylphosphonium halide, triphenylalkylphosphonium halide or tetraorganyl-ammonium halide.

A 10. A process as claimed in <sup>claim 1,</sup> ~~one or more of claims 1 to~~

A 20 ~~9,~~ wherein the temperature during the reaction with the yellow phosphorus is from -20 to +80°C.

A 11. A process as claimed in <sup>claim 1,</sup> ~~one or more of claims 1 to~~

A ~~10,~~ wherein the temperature during the reaction with the yellow phosphorus is from 0 to 30°C.

A 12. A process as claimed in one or more of claims 1 to  
A ~~11,~~ wherein the reaction is carried out under a  
pressure of from 0 to 10 bar.

A 13. A process as claimed in one or more of claims 1 to  
A 5 ~~12,~~ wherein the principal constituents of the  
mixture from a) are esterified in step b) using a  
linear or branched alcohol of the general formula  
R-OH, where R is a linear or branched alkyl radical  
having 1 to 10 carbon atoms.

A 10 14. A process as claimed in one or more of claims 1 to  
A ~~12,~~ wherein the principal constituents of the  
mixture from a) are reacted with mineral acids to  
give a mixture of alkylphosphonous, phosphorous and  
hypophosphorous acids and at the same time the  
15 (metal) salts of the mineral acids are precipitated,  
and the mixture of these acids is subsequently  
esterified.

A 15. A process as claimed in one or more of claims 1 to  
A ~~14,~~ wherein the water formed during the esterifica-  
20 tion is removed by azeotropic distillation.

A 16. A process as claimed in one or more of claims 1 to  
A ~~15,~~ wherein the alcohol is n- or i-butanol, n-  
hexanol, ethylhexanol and/or amyl alcohol.

A 17. A process as claimed in one or more of claims 1 to  
A 25 ~~16,~~ wherein the mineral acid is hydrochloric acid,  
sulfuric acid and/or phosphoric acid.

A 18. A process as claimed in one or more of claims 1 to  
A ~~17~~, wherein the mineral acid is hydrochloric acid.

A 19. A process as claimed in one or more of claims 1 to  
A ~~18~~, wherein the phosphines formed in small amounts  
5 in step a) are removed by oxidation.

A 20. A process as claimed in one or more of claims 1 to  
A ~~19~~, wherein hydrogen peroxide is employed for the  
oxidation.

A 21. A process as claimed in one or more of claims 1 to  
A ~~20~~, wherein the ester of the alkylphosphonous acid  
10 is removed by distillation in step c).

A 22. A process as claimed in one or more of claims 1 to  
A ~~21~~, wherein the ester of the alkylphosphonous acid  
15 is n-butyl methylphosphonite, isobutyl methyl-  
phosphonite, n-hexyl methylphosphonite, 2-ethylhexyl  
methylphosphonite and/or amyl methylphosphonite.

A 23. The use of an alkylphosphonous acid ester prepared  
by a process as claimed in claims 1 to ~~22~~ for the  
preparation of organophosphorus compounds and  
20 derivatives.

A 24. The use of an alkylphosphonous acid ester prepared  
by a process as claimed in claims 1 to ~~22~~ as a  
precursor for chemical synthesis.

A 25. The use of an alkylphosphonous acid ester prepared  
by a process as claimed in claims 1 to ~~22~~ for the

preparation of phosphinic acids as starting materials for crop protection agents.

26. The use of an alkylphosphonous acid ester prepared by a process as claimed in claims 1 ~~to 22~~ for the preparation of flame retardants.

27. The use of an alkylphosphonous acid ester prepared by a process as claimed in claims 1 ~~to 22~~ for the preparation of flame retardants for thermoplastic polymers, such as polyethylene terephthalate, polybutylene terephthalate or polyamide.

28. The use of an alkylphosphonous acid ester prepared by a process as claimed in claims 1 ~~to 22~~ for the preparation of flame retardants for thermosetting resins, such as unsaturated polyester resins, epoxy resins, polyurethanes or acrylates.